THE ANALYST.

APRIL, 1893.

PROCEEDINGS OF THE SOCIETY OF PUBLIC ANALYSTS.

The usual Monthly Meeting of this Society was held in the rooms of the Chemical Society, Burlington House, on March 1st, 1893. In the absence of the President, Dr. Dupré took the chair.

The minutes of the last meeting were read and confirmed.

The following gentlemen were proposed:—As Member, Mr. Walter Collingwood Williams, B.Sc. (*Honours in Chemistry and Physics, London*); as Associate, Mr. Hazlewood Irving Foster, assistant to Mr. James Baines, Hull.

Mr. W. Gathorne Hardy then read a paper on "A source of error in the Volumetric Estimation of Chlorides by Silver Nitrate Solution," which was followed by a discussion, and with this the Proceedings of the Society terminated.

ON THE USE OF CHRYSOTILE FIBRE IN PROXIMATE ORGANIC ANALYSIS.

By Thomas Macfarlane, F.R.S.C., Chief Analyst, Inland Revenue Department, Ottawa, Canada.

(*Read at the Meeting, December 7th*, 1892.)

Five years ago I read a paper on Milk Analysis before the Royal Society of Canada, which is published in their Transactions for 1887. The method there described involved the use of asbestos, or rather chrysotile fibre as an absorbent, and this substance has since been found so useful in the examination of other substances, chiefly foods, in the Laboratory of the Inland Revenue Department at Ottawa, that I believe a description of the manner of applying it will be found interesting to the members of the Society of Public Analysts. Of course, asbestos properly so-called has been used for many purposes by chemists, and especially for forming the filtering layer in the Gooch crucible. The fibre so used is frequently called Italian asbestos, and belongs to the mineral species, hornblende or actinolite. It is not changed by ignition, and is not nearly so flexible as the fibre derived from chrysotile. Chrysotile is the fibrous variety of serpentine, loses 12 per cent. combined water on ignition, and is then easily rubbed to powder between the

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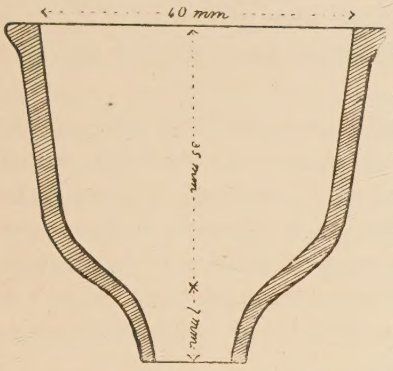
fingers. It is extensively mined in South Eastern Quebec, where it is found of a quality adapted for spinning and other finer manufacturing purposes. After being freed from rock and dust by grinding, sifting, and teasing, it assumes a woolly appearance; the fibres are silky and flexible, adhere to each other, and are quite destitute of the brittleness observed in glass wool, slag wool, or Italian asbestos. It cannot be called hygroscopic, for after long exposure to the atmosphere it contains only 0·57 per cent. moisture. The quantity used for an ordinary milk analysis is 2·5 grammes, which could only absorb by very long exposure 14 mgr. The change of weight during weighing must be therefore only infinitesimal. The chrysotile fibre is at present obtainable in London at the office of Messrs. Bolling and Lowe, 2, Laurence Pountney Hill, and it costs not more than 10d. per lb.

The use of chrysotile fibre as described in this paper is as a substitute for barytes, sand, powdered glass, calcined gypsum, &c., for absorbing and attenuating organic substances, so that their water may be conveniently removed and estimated, and so that the solvents used in their analysis may effectively permeate and act upon them. The advantage of the fibre is in serving this purpose in an eminent degree, while at the same time saving the chemist very much time, as well as trouble in manipulation. It is also to be remembered that the very complete drying effected by its means is of the greatest advantage in the operations to which the substance under examination may afterwards be subjected. I shall describe the manner of using it under the heads of the various substances, in the examination of which it has been successfully applied.

I.—Milk.

The chrysotile process of analysing milk has now been in use for over five years in the Inland Revenue Laboratory, as well as for a somewhat shorter time in that of the Department of Agriculture. It has been voluntarily adopted by all the public analysts in Canada, as well as by some in the United States. It has, of course, undergone slight modifications since first introduced, mostly for the purpose of saving time in treating a large number of samples. These are sometimes received at the rate of twenty or thirty a day in our laboratory at Ottawa, and I shall first describe the method as applied in this case. Samples are received from all parts of the Dominion, as far east as Sydney, N.S., and as far west as Regina in Assiniboia. They are always packed in ice, and arrive, as a rule, in good condition. Very likely, however, this packing in ice will be dispensed with in the near future, because on testing the method of preserving milk for analysis by introducing a small quantity of bichromate of potash, as proposed by Dr. J. E. Alén, of Gothenburg, I obtained very satisfactory results. This method will be found described in the *Deutsche Molkerei Zeitung* of 1892, pp. 224 and 416.

The glass tube used in the analysis, for containing the chrysotile fibre, has the shape and size shown in the figure. It is 40 mm. in diameter, 35 mm. in depth of bulb,



and 42 mm. in total depth to the end of the small tube. These tubes are made of well-annealed glass, and, after being numbered with a diamond and loosely filled with the chrysotile fibre, they are placed, to the number of 20 each, in copper trays or racks, measuring 9 inches by $7\frac{1}{4}$ inches, and having sides one inch deep, from which copper wires are stretched across, dividing the tray into 20 rectangular spaces. Four of these trays can be placed, one above another, in an ordinary box water bath, measuring $9\frac{1}{4}$ by $7\frac{3}{4}$ by 8 inches inside. The bath or oven we use is of this size, contains 80 tubes, and is supplied with a Soxhlet metallic back-flow condenser, so that it can be kept at work for days continuously. Two extra trays have been found necessary when the samples are sent in rapidly, and as many as 120 tubes have been in use at one and the same time.

After having been thoroughly dried, a tray with its 20 tubes is removed from the bath, and the tubes placed in a desiccator to cool. Each tube is then weighed and replaced on the tray. This weight, which includes both tube and chrysotile contents, I prefer to take in grains for the purpose of saving calculation afterwards. The quantity of milk used for each analysis is then run into the tubes as they stand in the tray from a 10 c.c. pipette which delivers exactly or very nearly 10 grammes. Of course the point must be ascertained by experiment up to which the pipette must be filled in order to deliver this quantity. It is usually about three-eighths of an inch below the ordinary mark when the pipette is allowed ten seconds to drain. The sample is thoroughly mixed, and the pipette washed out with it before filling each tube with the 10 grammes. Each sample is usually assayed in duplicate, and in this way 20 tubes representing 10 samples are quite rapidly filled. The samples have usually consecutive numbers and so have the tubes as they stand in the racks from left to right. By adhering to this system there is little or no risk of interchanging the samples or tubes. The chrysotile fibre rapidly absorbs the milk, and if the tube has been properly packed with it nothing escapes at the lower end. In the case of sour milk there is, however, danger of this, and in this case a larger quantity of fibre must be used, and the curd mixed with it by gently stirring the assay with the point of the pipette.

After filling the 20 tubes the tray is replaced in the water bath, and left there during the night—six or seven hours at a temperature of 96° C. are usually sufficient, but I prefer to keep the bath running the whole night and to dry at a lower temperature, say as low as 85° C., by leaving the door open to a slight extent. In this way it is possible to dry the milk thoroughly without any discolouration. It is in thus drying at low temperatures and in the saving of manipulation that the advantage of the process consists. The fine fibres insert themselves between the particles of the milk and facilitate the

escape of the water. It is not necessary to stir or touch the assay after the introduction of the milk, as is required in cases where sand, gypsum, or barytes is used for the same purpose as the chrysotile.

After drying, the tubes are cooled in the desiccator and weighed. In doing this the assay is placed in one scale, the tare in grain weights above-mentioned in the other, and then gramme weights are added to the latter until equal poise is attained. This weight in grammes multiplied by ten gives the percentage of total solids contained in the sample. As a rule it is about 0·3 per cent. less than when the assay is performed in a platinum capsule. The solids and fibre together form a rather tough mass in the tubes and in this perforations are made with a sharp-pointed instrument in order to facilitate the subsequent extraction.

The removal of the butter-fat from the contents of the tubes is effected in a large chloride of calcium jar, 18 inches high, whose upper part has an inside diameter of $1\frac{7}{8}$ inches, without any contraction at the top. The tubes are placed one above another in this upper part, which is 14 or 15 inches long, and capable of containing 12 or 13 of the assays. The tubulus in the lower part of the jar is then tightly corked, and the jar filled to above the level of the highest assay with petroleum ether, having a specific gravity of about 0·670, with boiling point ranging from 60 deg. to 85 deg. C. The jar is then closed tightly with a bung, and the whole set aside for two hours, during which time the assays are thoroughly permeated with the petroleum ether and most of the butter fat dissolved. The ether is then withdrawn by the tubulus at the bottom of the jar and the tubes allowed to drain. Fresh ether is then poured in upon the uppermost tube, when it percolates through all the other assays and collects in the receptacle at the bottom. After the latter has been filled four successive times by the ether thus used to wash away the butter fat, and all the ether has drained away from the tubes, the latter are freed still further from ether by drawing a current of air through them by means of the ordinary apparatus used for quick filtering. The tubes are then exposed in a steam bath to a temperature of 95 deg. or 100 deg., which completely frees them from the ether. They are then cooled in the desiccator and weighed, the loss being the butter fat. A much larger quantity of petroleum ether is used in this way than in working with the Soxhlet and other extracting apparatus. In our laboratory, however, a copper still heated by steam is used for recovering the ether, and the distillation of the extra quantity used occasions very little additional trouble.

Of course, the Soxhlet apparatus may be used as described in my paper of 1887, and much of our work has been done in this way. We have also made use of a Stutzer tube similar to that described in Böckman's "Untersuchungs Methoden" (edition 1884, page 579). Instead, however, of supporting the inside tube by means of a spiral wire, the Stutzer itself has three protuberances jutting inwards at its contracted part upon which the tubes containing the fibre and milk rest during the extraction. In such a Stutzer as

many as five or six tubes may be operated on at the same time. In an interesting paper read before the Royal Society of Canada in May, 1890, Mr. Frank T. Shutt, chemist of the Central Experimental Farm, has shown that the extraction of the butter fat is performed at least as effectually in these Stutzer tubes, and, in fact, his figures show that the Stutzers give slightly better results than when the extraction is performed in Soxhlets.

The analysis of milk in these tubes may be carried further than the determination of the butter fat, although the amount of the latter and the total solids is all that is usually required. The tube and its contents may be extracted with alcohol, and from the loss in weight the percentage of lactose ascertained. In this case, however, it is necessary to deduct from the result the weight of a small quantity of coagulum which separates from the alcoholic solution, and the ash yielded on evaporating the latter and igniting the product.

II.—Butter.

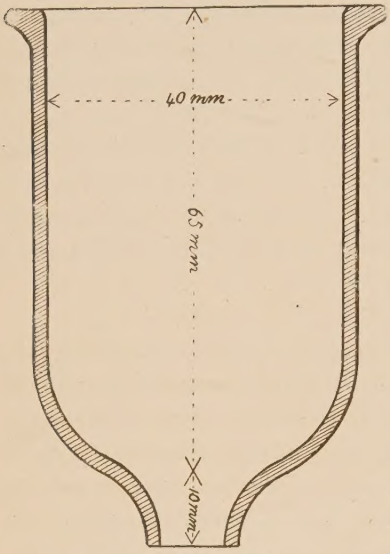
The application of the chrysotile fibre to the examination of butter concerns only the estimation of the water, fat, salt, and curd which it contains, and has no reference to the detection of foreign fats. Where the tubes or cups are to be used for butter the chrysotile fibre is placed in them in successive layers, so that the top one may be removed without disturbing the others. After a tube is dried and weighed, the top layer of fibre is removed, the butter to be examined introduced, and the top layer replaced carefully, so as to prevent any of its constituents from being removed by a possible splashing of the solvents. On weighing the tubes again the amount of butter taken for one assay becomes known. When the tube is placed in the water-bath, the butter melts, is absorbed by the fibre and the water is dissipated in the course of an hour. On cooling and weighing the tube, its amount is ascertained from the loss. By means of petroleum ether the fat is next dissolved out, and its amount obtained in the same way. Water next removes the salt, the curd is left insoluble, and from the next weighing the amount of both ascertained.

In heating the sample for an hour or two in the fibre in order to the dissipation of the water, there is not much reason to dread error from the oxydation of the butter fat. A sample exposed for seventeen hours in the water-bath, at a temperature of 90°C, only experienced an increase in weight of 0·82 per cent. calculated on the butter fat.

III.—Cheese.

The glass tube or tubulated cup which is used for the examination of cheese is of larger dimensions than that used for milk analysis. It has an outside diameter of 40mm., and a greatest length of 75mm., the depth of the wide part being 65mm. In packing it with the fibre it has been found advantageous to place a small piece of platinum or wire gauze over the small opening in the bottom to prevent the fibre from being drawn down into it when the contents are being subjected to the action of the filter pump. The fibre itself is introduced in successive loose layers, so that they may be removed and replaced singly when desired. After the tube with its contents has been dried and weighed, all

the chrysotile, excepting the layer at the bottom, is removed, and 5 grammes of the cheese in thin slices mixed or kneaded up with it, replaced in the tube and then covered over with the uppermost layer of the fibre. On weighing the whole, the weight of the cheese employed may also be again ascertained. The tube is then dried in the water-bath at a temperature of 98°, and a very perfect elimination of the water effected. This is proved by the close agreement of duplicate analyses. On cooling and weighing the tubes the loss in weight indicates the quantity of moisture present.



The tube is next subjected to extraction in a Soxhlet or Stutzer, and the loss which it experiences regarded as butter-fat.

The contents of the tube are next subjected to the action of warm water containing one per cent. of acetic acid. It is, in fact, washed therewith, while the tube is connected with the vacuum pump. The loss thus sustained is lactose, salt and soluble matter, the latter including, of course, the peptones and other soluble transformation, products of the caseine which have been produced during the ripening of the cheese.

That part of the cheese remaining as residue among the chrysotile fibre in the tube is regarded as unaltered casein.

IV.—Fats and Oils.

The amount of water contained in these, may, of course, be ascertained in exactly the same manner as described under butter, and this plan has been very successfully carried out in our laboratories in the examination of lards, some of which have been found to be adulterated with 5 to 25 per cent. of mechanically combined water. It the course of these analyses it was found that some lards, although heated for only a short time, increased in weight. Following up this observation, it was found that such lards were generally free from water, but adulterated with cotton seed oil. In fact, while pure lards exposed to a temperature of 95° C. for eighteen hours in chrysotile fibre seldom show a greater increase than 2·5 per cent., compound or adulterated lards always show over 3 per cent., and not unfrequently as high as 4·5. It is not supposed that this test would, taken by itself, be sufficient to condemn a lard or otherwise, but in conjunction with other data it has been found valuable. In cases where by a judicious admixture of beef tallow, lard, and cotton seed oil, the lard shows a satisfactory iodine number, it has been found possible to detect such adulteration by the use of the nitrate of silver test, in conjunction with the one now referred to.

It is quite possible that this method of determining the capacity which oils and fat have of absorbing oxygen may be found worthy of a wider application. I may mention the following percentages of increase in weight, which the following substances experienced on being heated for 18 hours in chrysotile fibre :—

Cotton seed oil	5·45—5·64 per cent.
Oleo oil from Chicago	0·37—0·44 „
Oleo stearine	no increase
Beef tallow, home rendered and filtered,	no increase
Mutton tallow	0·21—0·31 „
Porpoise oil	4·85—4·98 „
Neats foot oil	3·06—3·29 „
Olive oil (Barton and Guestier) ..	4·29—4·49 „
Linseed oil	4·72—5·04 „

Of course, these absorptions vary as the time of exposure is increased or otherwise. In the case of linseed oil the increase is much greater in the cold when plenty of time is allowed. Some very interesting results are obtained when oils are in this way allowed to attain the maximum degree of oxidation, and are then subjected to the action of various solvents, but these results are not yet complete in my hands, and should, at any rate, be treated of in a separate paper.

V.—Soap.

It must have occurred to some of the members now present that the same method of drying which I have described as applicable to cheese would also answer for soap, and such is the case. In several respects the use of chrysotile fibre is preferable to that of sand as described by Mr. Wilson in the *Chemical News* of 21st October last, p. 200. The soap may either be introduced into the fibre in the same manner as the cheese or after solution in alcohol. After drying at 98° C. closely agreeing results are obtained with duplicate samples.

The dried soap entangled among the fibre in the tubulated cup is then subjected to successive extractions, in Soxhlets or Stutzers, by the following solvents : petroleum ether, alcohol and water, the loss sustained by each treatment being noted. The first loss represents unsaponified fat ; the second soaps, free alkali and glycerine ; the third alkaline carbonates, salt, &c., while the insoluble substances, whether of organic or mineral origin are ascertained from what remains in excess of the original weight of the tube and fibre.

Of course the ethereal, alcoholic and aqueous solutions obtained in this way are all available for other necessary separations and determinations.

VI.—Spirits and Malt Liquors.

It is chiefly in the determination of the amount of extract contained in these that the chrysotile has found application. The result has been a more perfect elimination of the

water, and a decrease in the percentage of the extract. The following statement shows the differences between this and the ordinary method of determining the extract in such beverages :—

	Grammes Residue per litre when dried at 100° C.		Percentage of additional loss when dried in chrysotile fibre.
	In Platinum Capsule.	In Chrysotile Fibre.	
Champagne Cognac, 1865	16·654	14·746	11·46
Hennessy's Cognac V.O.	9·180	8·520	7·19
Gooderham & Wort's Rye Whisky ...	1·959	1·842	5·97
Canadian Porter, Walkerville B. Co.	55·200	53·400	3·26
Allsopp's Red Hand Ale	56·900	55·475	2·51
Brading's Ale, Ottawa	37·050	36·650	1·08

With reference to the amount of extract contained in worts and de-alcoholised beer, it is admitted by most authorities that the percentages given in the Schultz tables as corresponding to certain specific gravities are too high. Schultz obtained his figures by determining the specific gravity of various worts at 15° C. and then evaporating to apparent dryness at from 75° to 80° C. In this way it is not possible to obtain an extract free from water, and recognising this, Dr. A. Elion constructed in April, 1890, a new set of tables (*Zeitschrift für angewandte Chemie*, 1890, p. 291) based upon experiments in which the extract was determined by drying in a rarefied atmosphere at 97° C. These values are much lower than those of Schultz, but it is not certain that even they show the amount of dry substance perfectly free from water. Our method of drying in chrysotile at 92° C. gives even lower results and in the following table are given some of those obtained in a series of determinations of de-alcoholised beers as also, for the sake of comparison, the specific gravities and the amount of extract corresponding thereto according to Schultz, to Elion, and to the saccharometer tables of Bates :—

No. of Sample.	Description of Sample.	Sp. gr. of spent beer 15° C.	Grammes solid in 100 c.c. according to			Grammes solid in 100c.c. dried in chrysotile fibre.
			Schultz.	Elion.	Bates.	
4.	Sparkling Amber Ale	1·02191	5·79	5·40	5·55	5·413
9.	Hamilton's London Amber Ale	1·01194	3·11	2·96	3·06	2·801
10.	Dörbecker's Lager Salem	1·02044	5·41	5·09	5·06	4·070
16.	Dow's India Ale	1·02148	5·69	5·36	5·31	4·925
18.	Toronto Brewing Co. India Pale Ale	1·01190	3·11	2·96	3·06	2·625
20.	Carling's India Pale Ale	1·01709	4·50	4·26	4·31	3·940

From these figures it will be observed that as stated above, the results obtained by drying in chrysotile fibre are invariably lower than even Elion's figures, although in some cases the difference is very slight. Elion's experiments were made with genuine malt extracts and beers, and it is still uncertain as to whether these differences are owing to the method of drying, or to something abnormal in the beers above mentioned.

In order to ascertain the nature of the constituents of these beer extracts, the tubes containing them and the fibre are subjected to extraction by alcohol of 96 per cent. (by volume). Sugar which has escaped fermentation and other substances are thus removed, and the tubes, after drying, &c., are weighed. The substances remaining in the chrysotile fibre are albuminoids and dextrin. A nitrogen determination on the original beer by the Kjeldahl method enables the quantity of the former to be calculated, and on its subtraction from the amount of residue the quantity of gum and dextrin becomes known. This is an important figure which renders it possible, in conjunction with the degree of fermentation (Vergärungs grad., *i.e.*, the percentage loss of original constituents in the wort during fermentation), to determine whether saccharine substances have been used in the brewing.

VII.—Syrups and Molasses.

The difficulty of drying these products is well known, but it is overcome by the use of chrysotile fibre. The quantity used for analysis must not, however, be introduced direct among the fibre. It must be diluted with four or five times its weight of water, and then an aliquot part of the solution may be pipetted into the tube among the fibre. After drying these for 48 hours, there is no difficulty in obtaining concordant results.

The tubes thus dried may be extracted with alcohol and the solution used for the saccharometer. I believe more correct results are thus obtained than by direct polarisation; but to discuss this matter would go beyond the limits of this paper.

VIII.—Coffee.

Not only has it been found possible to treat with advantage liquids, oils, and viscous substances in chrysotile fibre, but it has also been used for enveloping and enclosing powdered substances, in place of the usual capsules of filtering paper, while they are being acted on or extracted by various solvents. One of the most important of these is coffee, regarding which I shall venture to go more into detail. The tube in which it is treated is the same as that above-described under the heading "Cheese." The manner of filling it with the fibre is the same, and care must, of course, be taken, in this as in other cases, that it is well teased out and not packed too closely in the tube. Owing to the large number of samples which are usually delivered to the laboratory at one time, the tubes are arranged in copper racks holding 20, all properly numbered, and are dried for 24 hours in the water oven at 100 degs. C. The tubes are weighed from the desiccator and immediately returned to it. After their weight is known they are each charged with from 5 to 7 grammes of the coffee to be analysed, which has been previously ground to a standard fineness. The filling is performed by rapidly removing the chrysotile layers with metal pliers, over black glazed paper, and replacing them with a layer of, say, 3 or 4 mm. of coffee between each. A very thick plug or layer of fibre is placed on the top, and the loaded tube is immediately weighed to ascertain the exact amount of the sample taken.

After 24 hours in the water oven at 100 degs. C., the weight is again taken, and the loss is stated as moisture.

Curiously enough, this loss is somewhat greater than when the coffee is dried on thin layers on watch glasses. In 18 samples dried by both methods simultaneously, the following numbers were obtained:—

Sample.	Loss at 100 degs. C. in 18 hours. On Watch Glasses.	In Chrysotile Fibre.
1.	6·73	7·07
2.	6·13	6.44
3.	4·20	4·51
4.	5·65	6·11
5.	5·28	5·58
6.	9·84	9·80
7.	10·39	10·69
8.	3·83	4·70
9.	5·27	5·65
10.	5·71	6·01
11.	5·12	5·32
12.	4·74	5·01
13.	6·12	6·42
14.	5·39	5·64
15.	4·08	4 51
16.	6·80	7 14
17.	4·78	5·08
18.	4·86	5·04
Mean	5·82	6·15

The above are samples of commercial coffee, some of them very much adulterated. The loss of weight is practically constant at 18 to 20 hours. In 20 tubes, containing an average of 6·133 grammes each, the loss of weight in 20 hours averaged 262 millegrammes, and for the next 24 hours a further loss of only 3·9 millegrammes occurred.

The tubes are now exhausted by petroleum ether, which may be done either in Soxhlet or Stutzer tubes, two or three of the assays being treated at once. A very large number of samples have been worked over in this way in duplicate, with very satisfactory results. Genuine coffees yield from 10 to 12 per cent. of fat by this treatment. One genuine sample gave only 8·20 per cent., but, in this respect, stands alone among nearly 50 pure coffees. No sample gave as much as 13 per cent., although 12·5 has been reached in a few instances. Several of the district analysts have obtained higher figures, but this is owing to the use of ethylic ether in the extraction,

instead of petroleum ether. The quality of the latter used in the Inland Revenue Laboratory is obtained by distillation from "deodorised gasoline" at temperatures ranging from 60° to 85° C. The percentage of fat contained in adulterated coffee varies from 12 to 2 per cent., and as a general rule it may be assumed that the lower the percentage of fat the greater is the amount of adulterants in the coffee. It would, however, be unsafe to depend entirely on this, as there is nothing to prevent the manufacturer from introducing foreign fat into the mixture if he finds it to his interest to do so. Chicory gives about 1 per cent. of extractive matter to petroleum ether, and it may be added here that three samples of roasted barley yielded respectively 1·31, 1·54, and 1·42 per cent. to the same solvent. All the percentages of fat extracted are calculated on the dry substance.

The tubes are next treated with boiling water in order to determine the amount of water extract. They are placed in the rack and boiling water poured into them, and after standing about 15 minutes they are placed on the jars connected with the filter pump, in the same manner as for the washing by ether, and repeatedly washed with boiling water until the washings have but little colour. This requires an average of 10 minutes for each tube, and about 250 c.c. of water. On being dried as thoroughly as possible by the pump, they are returned to the oven and kept at a temperature of 100° C. for 48 hours. The loss of weight is the water extract, and amounts to about 22 per cent. in the case of genuine coffee. Chicory gives nearly 80 per cent. (77·73 per cent. according to our experiments), and it has been proposed to make use of the percentage of water extract obtained in this way as a substitute for the indications of the ten per cent. decoction in the cases of mixtures of coffee with chicory only. In the case of samples containing other adulterants as well, the amount of water extract varies from 25 to 68 per cent., the high figures being occasioned by the sugar, caramel, gum, soluble starch, and dextrin contained in the substances which are mixed with the coffee in order to produce a cheap article.

The foregoing descriptions do not by any means exhaust the list of substances whose examination or analysis has been facilitated by the use of chrysotile fibre and the various glass tubes mentioned. Mustard may be treated in exactly the same manner as coffee, and its moisture and fixed oil contents conveniently determined. Pepper may also be distributed among the fibre, and the moisture and alcoholic extract determined. I have also introduced common gunpowder among the fibre in these tubes, and by successive extractions with carbon disulphide and water determined its percentages of sulphur, nitre, and carbon. The examination of varnishes is much facilitated by driving off the volatile constituents in these tubes and then subjecting the residue to the action of various solvents. Mr. Shutt informs me that he has conveniently and successfully performed the analysis of condensed milk by using this system, and I have not the slightest doubt that the water contained in honey could be easily eliminated and estimated by the use of chrysotile fibre. It has also seemed to me that it, in conjunction with the tubes for containing it, constitutes

the best possible means for carrying out to a considerable extent the plans described by Dragendorff and others in the analyses of plants. I sincerely trust that my fellow members of the Society of Public Analysts will give these methods and suggestions some consideration, for I feel convinced that their adoption in whole or in part will materially lighten their labours and economise their time.

Although I have to bear the chief responsibility of introducing the use of chrysotile fibre into the methods of the Inland Revenue Laboratory at Ottawa, I must not neglect to state that much of the work referred to in this paper has been performed by my assistants, Mr. A. McGill, M.A., Mr. F. W. Babington, F.I.C., and Mr. A. L. Tourchot; and I take this opportunity of gratefully acknowledging the interest, zeal, and ability with which they have worked out some of these problems in proximate organic analysis.

Discussion.

The President said that Mr. Macfarlane had dealt with a great variety of subjects in his paper. It was only quite recently that Mr. Richmond had again brought before the Society the system of evaporating milk by means of asbestos wool, which he had been experimenting with. No doubt it was unfortunate that public analysts, something like fifteen years ago, at the instance of Mr. Wanklyn, had ceased to use the method which was until then universal, of evaporating milk with diluting substances like sand or pumice. He was surprised at one or two remarks made by Mr. Macfarlane. He (the President) would have expected that with the use of the crysotile fibre he would have obtained higher total solids than without it, for the reason that, if such a substance as milk, or anything of a sugary nature were dried *en bloc* in a platinum basin, it was excessively difficult to dry, in fact it was almost impossible. Milk, when evaporated, always got more or less discoloured, whilst Mr. Richmond did not find any such discolouration by the use of asbestos fibre for evaporating milk, and he (Mr. Richmond) obtained high results. In the case of wine and malt wort, the figures for total solids obtained by evaporation, as proved by Dr. Dupré a good many years ago in the case of wine, were always smaller than those obtained by the specific gravity of the de-alcoholised liquid. Dr. Dupré had pointed out that a certain amount of dehydration took place, which altered the character of the residue. The observation as to the oxygen absorbed by oils would be a very great help, especially in testing for cotton seed oil. Cotton seed oil was now so treated, with a view to deceiving analysts, that the old silver test sometimes appeared to fail. He had had a number of samples of lard which he was almost convinced contained cotton-seed oil, or some very similar oil. If a corroborative test could be found, it would be exceedingly useful. At the same time the oxygen absorption test for oils had been used for a great many years in examining many forms of oils.

Mr. H. Droop Richmond said he would like to state, in reference to the President's remark, that he had not claimed the asbestos method as his own. In his paper (Analyst

xvii., 225) he had mentioned that some years ago the asbestos method had been introduced in America by Mr. Macfarlane or Mr. Babcock. Would Mr. Macfarlane tell the meeting whose method was first published.

Mr. Macfarlane replied that there could be no doubt that Mr. Babcock first introduced the use of asbestos, but he thought that Mr. Babcock did not introduce the use of crysotile fibre or the method of obtaining the results by difference. He did not bring forward this method as anything particularly new, except in the manner of applying it so that the butter fat could be determined by difference, and a much larger number of samples analysed than the analyst otherwise could deal with in the same time.

Mr. Richmond wished to ask Mr. Macfarlane how the results for fat agreed with the ordinary methods; that is to say, with the extraction of the fat by the Adams or Werner-Schmid processes. He (Mr. Richmond) wished to say that he shared the President's surprise at the results of Mr Macfarlane's determinations in the case of milk, being below those given by the ordinary method. The results by the asbestos process he had found, if anything, a little higher; and on drying for a very considerable number of hours—up to, in some cases, close on 60—he had found that the weight remained practically constant; sometimes it had gone half a milligram up or down. He could not get it to lose any more. He (Mr. Richmond) put the asbestos in a platinum basin and ignited it, and all the water in the asbestos, whether it was condensed moisture or whether it was combined water, was driven off. Mr. Macfarlane had stated that he sometimes had his milk samples sent him in ice. He (Mr. Richmond) was strongly of opinion that it was a bad thing to put milks in ice. It caused separation of fat in such a way as to render proper admixture afterwards difficult. He preferred to have his samples kept at a warmer temperature, even at the risk of their curdling sooner.

Mr. Macfarlane did not think that this method had been compared with any other than that of ordinary evaporation in platinum vessels, and certainly the loss was greater in crysotile fibre than it was in platinum.

The President asked whether the total solids were determined by drying to constant weight, or by the usual process of drying for three hours?

Mr. Macfarlane said that it was possible to dry the milk in four or five hours at a temperature of 98° C., but he preferred to leave the samples in the water-bath over night at about 92° C. In determining the solids in platinum, and comparing the results with those obtained in crysotile fibre, the same time and temperature was employed. There could not be any doubt that more water was eliminated by using crysotile fibre. As to any loss the crysotile fibre might sustain when dry, it had been exposed to temperatures under and up to 100°C. without its losing weight. This variety of crysotile loses 12 per cent. combined water on ignition. In this case it loses its character altogether, and falls to powder. With regard to Mr. Richmond's question, no other plan could be devised

than packing it in ice ; unless, indeed, the method of Dr. Alén, of Gothenburg, by bichromate of potash, should be found to answer, when the milk had to be sent long distances. When the milk arrived cold enough he had never had any difficulty in consequence of the separation of the cream.

Mr. Richmond wished to ask Mr. Macfarlane how the results under this method compared with the Adams and other processes ?

Mr. Macfarlane did not think that he had made such comparisons. He did not use the Adams method. There was so much manipulation connected with it that the large number of milk analyses required could not possibly be got through in the proper time. He had to adopt some other method whereby the manipulation was not so great. The actual variation had been determined by Dr. Ellis, of Toronto, between this and the Adams method, with the practical result of his adopting it instead of the Adams ; not that it was considered more correct, but because a large amount of time and trouble was saved. A good deal of work had also been done by Mr. Schutt, of the Experimental Farm Laboratory at Ottawa, in these comparisons, and he (Mr. Schutt) had always found a less quantity of total solids by using the fibre than otherwise.

Dr. Muter said that as time rolled on they returned to the old forgotten methods Mr. Macfarlane's process was practically similar to the one that he (Dr. Muter) practised twenty-seven years ago, when he first made a speciality of the analysis of food and drugs. It was a very good method, because it gave the actually dry residue ; and was only abandoned in deference to the general agreement to adopt the Wanklyn method, as to which he agreed with the President's remarks. In the old way a small funnel, with a glass cover, was plugged, nearly filled with sand, dried in a specially constructed bath, and weighed. The milk was run on to the sand, and the whole again dried and weighed. The funnel and its contents were then percolated with ether, and the fat thus removed weighed. The residue was then percolated with warm dilute spirit, to remove sugar (estimated by Fehling) ; and finally the sand was dried and submitted to combustion with soda lime for the proteids. Then nothing was known as to the constancy of the solids-not-fat, and consequently full analyses were always made. He merely mentioned this as a matter of curiosity, but even now it was not a bad way of attacking condensed milks, which contained an abundance of cane sugar. As a rule, with the sand, or other similar method, the total solids came 0·1 to 0·2 below those taken by simply drying in a dish. For ordinary commercial work he considered that no process was so good as the solids, gravity, and Richmond's slide rule.

Mr. Macfarlane in compliance with a request made by the President, then gave some particulars as to the labours of Public Analysts in Canada. The laboratory branch of the Inland Revenue Department was charged with the administration, not only of the Adulteration Act, but of the Fertiliser's Act, and he would try to state how this was carried out. There were eight district analysts in the Dominion, one in each of the cities

of Halifax, St. John, Quebec, Montreal, Ottawa, Toronto, London, and Winnipeg. Those gentlemen were professional men, of course, and prepared to do all the analytical work the public had to offer them. They were also appointed public analysts by the Government, and in order that they might take their part in carrying out the Adulteration Act, they were supplied from time to time in the course of the year with a certain number of samples to analyse. These samples were collected by food inspectors, of whom there were as many as there were analysts, that is one for each district. The instructions received by the food inspectors, were issued by the laboratory branch after the Commissioner of Inland Revenue having been consulted as to what articles should be analysed. Orders were issued to the food inspectors to collect so many samples of foods, drugs, or agricultural fertilisers, some perhaps of a particular kind, and to deliver them to the public analysts. The food inspectors went into the shops and places of business and asked, as if they were ordinary purchasers, for the articles in question. According to the law, and according to the practice in Canada the vendor was then and there, after the purchase had been made, informed of the purpose for which the sample was obtained. The sample was then divided into three parts, each sealed up, one left with the vendor, one sent to the district analyst, and the remaining one to the departmental laboratory at Ottawa. The next step was the analysis by the district analyst of the sample and his report to the Commissioner of Inland Revenue. The analyst was paid certain fees on the delivery of his certificate showing whether it was or was not adulterated. The certificates were then inspected by himself (Mr. Macfarlane), and the results placed on record in the books kept by the branch at Ottawa which were properly systematised. He was able with the aid of the laboratory staff, to go through all the samples which had been analysed by the district analysts and check their results, but as a rule only those found by them to be adulterated were considered to be worth revision in this way. In such cases the sample sent to Ottawa was analysed in the laboratory there, and if the results obtained by the local analyst were confirmed, the next proceeding was to "apply the Act," carrying out its provisions in proper order. Of course they could at once report certain cases to the Department of Justice for prosecution, but the Act gave them the power of previously intimating to the vendor that his sample had been found to be adulterated: and they had also the power under the Act of calling upon him to pay the cost of collecting and analysing the sample, which cost varied from eight to sixteen dollars. The vendor could elect to pay the fine, or to refuse and stand a prosecution. This plan was adopted, and an amendment of the Act was inserted to that effect, because it was found that in a great many cases, if the vendor were prosecuted, and a conviction was obtained, the resulting fines were far from adequate to pay the expenses of the prosecutions. The consequence was that the grant of money for carrying out the Act which was given by the legislature, had to stand large outlays for legal expenses. On this representation being made to the Minister of Inland Revenue he suggested the method now in force for overcoming the difficulty. In three-

fourths of the cases the fines were paid without any prosecution being necessary, the vendor preferring to pay, rather than to be subjected to a prosecution. The names of those who did not pay, were given in to the Department of Justice, and a prosecution took place in the ordinary way. In law there, as here, there was much uncertainty, but in the majority of cases convictions were obtained. He could only remember that they had failed in the case of baking powder, the lawyers raising the question as to whether that was, or was not a food. In addition to such legal proceedings, bulletins were published in which the names and addresses of persons from whom samples had been taken were given, and also the results of the examinations, both of the genuine and adulterated articles. The publication of these bulletins was quite anxiously looked for by the public, and the Press readily took notice of them. The local newspaper, too, was sure to publish the names of those individuals in their neighbourhood who had been found to sell adulterated goods; so that, on the whole, during the last six years it could fairly be said that the branch had done a great deal of good, and had done much in the direction of improving the quality of the articles sold throughout the whole Dominion. They also had the support of the public, and, to a very large extent, of the dealers also, as well as the manufacturers. It was merely a question of getting the Act applied to a sufficient extent. The difficulty was that, notwithstanding, a good many adulterators escaped. If the Act were administered in its entirety, and all of its stringent provisions applied, the officers of the branch might possibly make nuisances of themselves. Their powers were so great that they might end by bothering everybody. Care had to be taken that unreasonable prosecutions were not instituted, and as long as this was done they would have the support of the public and the traders. The bulletins before the meeting showed the way in which the names were published, and it would be seen how each article was treated.

The President asked Mr. Macfarlane what he considered reasonable limits with regard to milk. What course did he adopt with those samples which gave results only very slightly below the average?

Mr. Macfarlane replied that such cases were left severely alone. They certainly would prosecute in every case where the fat was less than 3 per cent.—even slightly less; anything below 3 per cent. would be condemned. The bulletins showed the judgments that had been given. He did not think he could at the moment pick out the doubtful samples which had been referred to by the President. He had sometimes cases of comparatively rich milk which, he was tolerably certain, had been watered; but as there were plenty of worse cases to be attended to, those were put down as "below average." It had been found by the analysis of a great number of samples of genuine milk that the average percentage of fat in Canadian milk amounted to 3·75. Perhaps not more than 1 in 100 would be found to contain less than 3·45 per cent. of fat. They would be called "under average," although they were genuine. They (the analysts) were perfectly justified in

calling them "below average." What he had proposed was that the lowest limit should be 12 per cent. of total solids, of which 3·5 should be milk fat, and 8·5 other solids. No standard had been established by the Government in connection with this recommendation. The Act gave the Government the power to fix the limits of variability in the case of any article, but they were unwilling to do so unless there was a very great necessity for it. He did not think that his branch was impeded in its operations because there was no legal standard. Any milk containing less than 8·5 per cent. of non-fatty solids, or any milk containing less than 3 per cent. of fat, was condemned, and a prosecution was instituted, with a very good chance of obtaining a conviction.

Mr. Cassal asked Mr. Macfarlane whether he would calculate the percentage of fat abstracted on a limit of 3·5 per cent. of fat, or of 3·0 per cent. of fat.

Mr. Macfarlane stated that the adulteration branch would not prosecute for anything between 3·0 and 3·5, but that it would do so if the sample contained less than 3 per cent.

Mr. Cassal explained that he wished to know whether Mr. Macfarlane would report that a particular sample of "milk" deficient in fat had had a certain percentage of its original fat abstracted, and, if so, whether he would calculate that percentage on the limit of 3·5 per cent. of original fat, or of 3 per cent. He gathered that Mr. Macfarlane reported analytical data, namely, percentages of solids, fat, and so forth in his certificates, or in the bulletins, a course which he (Mr. Cassal) was glad to say had now been given up by most of the leading English public analysts; but he supposed that when cases of adulteration went into Court the meaning of the analytical data was given in the form of percentages of added water or of fat abstracted.

Mr. Macfarlane stated that it was not necessary to state the solids, fat, &c., he only went as far as was necessary to obtain a conviction.

The President wished to know, supposing Mr. Macfarlane, as head of the department, had analysed a sample which the district analyst had reported as adulterated; and supposing that the two results happened to differ, whose opinion would prevail? There were two ways of differing—either through the existence of an error, on one side or the other, or through a difference in the opinion founded on the analytical results. One chemist might arrive at the same analytical results as another, but he might draw different conclusions and give a different opinion. A case had occurred quite recently in London where a sample of milk had been analysed by three persons. The first said the milk was skimmed; the second said that it was both skimmed and watered; and the third said that the sample might have been genuine; while the results obtained by each did not differ very widely. If such a case occurred in Canada, how would it be dealt with, and what would be the consequences?

Mr. Macfarlane could only conceive of such cases occurring in the following

manner. As he had stated, the vendor retained part of the sample sealed up, and he was at liberty to employ any chemist to examine that sample, but during the whole of the six years that he (Mr. Macfarlane) had been in charge of the branch, he had not known a case where this sample had been referred to a private analyst. The vendors could refer them to the chief analyst, but they did not, as a rule, go to that trouble. In a few cases the vendors had sent their samples to Ottawa, and in one case his department found that an interchange of samples had taken place. When the vendors referred their samples to headquarters, they usually accepted the result without question. In the event of there being any difference of opinion between the chemists at Ottawa and the district analyst, it was usually found that by consulting together differences could be explained, No pressure was used by the department. The district analysts were perfectly independent; but it was quite within the province of the chief analyst in any particular case to suggest that the opinion of a district analyst was not quite correct, and to specify his reasons for the suggestion, and there were instances in which the certificates had been modified accordingly. It was not thought necessary that such differences of opinion should be made public; the whole matter was arranged privately. Everything was done to prevent injustice to the traders of the Dominion; but at the same time when prosecutions became necessary the analysts were perfectly united. It never happened that they gave opposing opinions in court; they would not allow such to become public. If differences existed which could not be reconciled, the matter was dropped, and no prosecution was attempted.

On the Determination of Ferric Oxide and Alumina in Mineral Phosphates **G. Mariani and E. Tasselli.** (*Staz. Sper. Ag. Ital.*, xxiii., 31.)—The authors show that Gläser's alcohol method has the following defects :—

(i.) The precipitation of a small quantity of calcium phosphate with the ferric and aluminium phosphates.

(ii.) The possible precipitation of basic phosphates, if all the iron and alumina are not in the mineral combined with phosphoric acid.

(iii.) The partial solubility of ferric and aluminium phosphates in dilute acetic acid.

(iv.) The decomposition of ferric ortho-phosphate by boiling water into soluble acid phosphate and insoluble basic salt.

They propose the following method :—1 to 5 grams of the phosphate are boiled in a flask with 15 c.c. strong hydrochloric acid for 10 minutes, and are then diluted with twice the volume of water; a few crystals of potassium chlorate, and several drops of nitric acid, and the liquid boiled to drive off the excess of chlorine. This is filtered, the filtrate and washings measuring about 150 c.c.; to the cold liquid a solution containing half a gram of ammonium phosphate is added, 2 c.c. of glacial acetic acid, and then

dilute ammonia drop by drop, until a small permanent precipitate remains; 2 c.c. of acetic acid are then added, and the liquid, after repeated shaking, is left for a couple of hours. The precipitate is collected on a filter, and washed with 1 per cent. ammonium phosphate solution.

The funnel is placed over the flask in which precipitation took place, and the precipitate is dissolved by a minimum of hydrochloric acid (sp. gr. 1·12), and a second precipitation is performed as before. The precipitate is well washed with the ammonium phosphate solution mentioned above, the filter dried, and burnt at a dull-red heat.

Half the weight of the residue is taken as ferric oxide and alumina.

A Thomas slag, two mineral phosphates, and an artificial mixture containing 1·60 per cent. of ferric oxide and alumina were analysed.

	New Method.	Greatest difference of duplicates.	Gläser's Method.	Greatest difference of duplicates.
Thomas slag	19·12	0·31	19·48	0·30
Mineral phosphate ...	4·01	0·45	3·72	0·25
„ „ ...	1·88	0·15	1·76	0·21
Artificial mixture ...	1·574	0·040	1·514	0·100

The authors do not consider their method perfect, but they put it forward as having many advantages. H. D. R.

Some Points in the Determination of the Intensity of the Colour of Wines, and in the Determination of Free Tartaric Acid. L. Sostegni. (*Staz. Sper. Ag. Ital.*, xxiii., 10.)—The author shows that when comparing the colour of wines with that of a standard solution of fuchsine (0·05 grs. per litre) the results vary very largely according to the length of the column used in the comparison. Duboscq's colorimeter was used. He also shows that different specimens of fuchsine have very different intensities of colour.

From his experiments on malic acid, he concludes that acid potassium malate is precipitated along with acid potassium tartrate in alcoholic and alcoholic-ethereal solutions unless 5 c.c. of water be present for each 0·1 gram of malic acid. If the precipitation takes place in the presence of acetic acid no malate is found with the tartrate. Sugar and other organic matter in wine hinders the precipitation of acid malate. H. D. R.

On the (so-called) Pennetier's Method for Butter Analysis (Microscopic Examination by Polarized Light). A. Pizzi. (*Staz. Sper. Ag. Ital.*, xxiii., 38.)—The author points out that previous experimenters, including himself, had used simple

mixtures of butter and margarine (*c.f.* ANALYST XVII. 193.); he now studies various conditions which occur in practice, in order to see if they modify the results obtained.

He finds that keeping butter for some time at a temperature (28°–29° C.) but slightly below its point of fusion, does not modify the results.

The semi-fused portions around the section made by a warm knife gave somewhat doubtful indications.

Butter exposed to the rays of the sun, and melted by this means, behaved similarly to butter fused by heat.

Various substances were added to butter; of these—

Boric Acid	gave	Suspicious indications.
Borax		,, ,,
Cane Sugar		,, ,,
Tartaric Acid		,, ,,
Salicylic Acid	,,	Crystals of colour different to field (with selenite plate.)
Sodium Salicylate		
,, Bicarbonate ...		
Milk Sugar		
Glucose		
Shavings of horny substance	,,	Grains and fragments red, some showing two or three colours.
Starch		Play of colours.
Saffron	,,	The characters of genuine butter not altered.
Annatto		
Turmeric		

A quantity of milk skimmed by Laval's separator was mixed with a quantity of margarine by Jespersen's Emulsor; this was mixed with cream and separated, and was then churned into butter, which contained about 30 per cent. margarine; this mixture behaved as genuine butter.

Freshly made, unwashed butter, butter kept under water for five months, and butter prepared from clotted cream all gave results as for genuine butter.

Rancid butter covered with fungoid growths behaved as margarine; rancid butter free from fungus appeared as margarine, when the outer portion was examined, and genuine when care was taken to use the inner part.

The author concludes that the method is sufficiently exact to prove the presence of margarine mixed with butter, but it is far from being of universal application on account of the many exceptions. H. D. R.

The Direct Determination of Nitrogen in Nitrates. A. Devada. (*Chem. Zeit.*, 1892, xvi., 1952.)—The method of reducing nitrates to ammonia by treatment with powdered metals, such as zinc and iron, in alkaline solution, is of considerable

antiquity, and gives good results if conducted sufficiently slowly. The same is true of Stutzer's method, in which aluminium foil is used, the rate of attack being slow in the dilute caustic lye that is employed. This inconvenience has been overcome by the author by the use of an alloy of aluminium, copper and zinc, which is easily powdered and has a powerful reducing action. The alloy, which can be obtained from T. Srpek, of Vienna, contains 45 per cent. of aluminium, 50 per cent. of copper, and 5 per cent. of zinc, which in dissolving leaves the copper in a finely divided state, so that it ensures regular distillation without bumping.

The process is carried out as follows: The solution, containing the nitrate in quantity equivalent to about 0·5 grams of potassium nitrate, is placed in a flask having a capacity of about a litre, diluted with 60 c.c. of water and 5 c.c. of alcohol, and 40 c.c. of caustic potash solution of sp. gr. 1·3 added. 2–2·5 grams of the alloy described above are introduced, and the flask attached to a condenser with a receiver containing standard acid. The connection between the flask and the condenser is made by means of a tube having on the limb next the flask a bulb filled with glass beads to prevent the contents of the flask splashing over into the receiver, and on the other limb another bulb to prevent the acid in the receiver finding its way into the distillation flask, should regurgitation occur. When the flask has been thus connected with the condenser it is gently heated for half-an-hour, at the end of which time the evolution of hydrogen will have slackened or ceased, and then the distillation is begun, at first cautiously, until the zinc of the alloy has completely dissolved, and then more vigorously, the time necessary being about twenty minutes from the time when the contents of the flask begin to boil. The distillate is caught in standard acid and the ammonia determined by backward titration in the ordinary way, It is to be noted that the strength of the alkali used is of importance, as if it be too strong, the action on the alloy is unduly vigorous at the beginning of the operation, and if too weak the contents of the flask have to be heated overmuch, the result in both cases being the formation of a fine spray of caustic solution, which is very difficult to stop, even with complicated attachments to the distilling flask. The test analyses on pure nitrates are satisfactory. B. B.

Qualitative Examination of Iodine for Cyanogen. C. Meineke. (*Zeits. anorgan. Chem.*, 1892, II. 165, through *Chem. Zeit.*)—According to the German Pharmacopœia, cyanogen can be detected in iodine by rubbing 5 grams of the sample up with water, filtering and reducing the solution with sulphurous acid, adding a very small quantity of ferrous sulphate and a little caustic potash, warming the solution, and, after cooling, acidifying it. The formation of Prussian blue indicates the presence of cyanogen. Sodium thiosulphate may be substituted for the sulphurous acid if the iodine solution be first acidified. One part of iodine cyanide in 12,000 parts of water, or one part of cyanogen in 54,000 parts of water can be recognised by this test. The behaviour of

iodine cyanide in neutral solution supplies two other methods for detecting it. A few milligrams of KI are added to a neutral solution of iodine containing cyanogen, which is then decolorised with sodium thiosulphate solution of about centinormal strength, until the yellow colour has almost disappeared. Clear starch solution is added, which, if iodine cyanide be present, produces a much deeper blue colouration than if the iodine be pure. The object of the potassium iodide is to obtain the full blue colour with starch. The reappearance of the blue colour of the iodide of starch, but with a redder tinge than is normal, is also an indication of the presence of cyanogen. The other method depends on the formation of sulphate when iodine cyanide is reduced with sodium thiosulphate, which can be detected by the addition of barium chloride in the usual manner. The freedom of the thiosulphate from sulphate must be ascertained by a blank experiment with pure iodine. The reaction is about twice as sensitive as that obtained by the official method.

B. B.

[The official method, which is absolutely characteristic of cyanogen and depends on no indirect reaction, is, nevertheless, to be preferred.—B. B.]

The Determination of Ash in Mineral Lubricating Oils. A. Stepanow. (*Zap. imp. russk. tech. obszcz.*, 1892, xxvi., 927, through *Chem. Zeit.*)—The direct incineration of mineral lubricating oils is inconvenient, as they evolve much soot and leave a coke difficult to burn off. The plan adopted by the author consists in the use of a head fitted as closely as may be to the dish in which the oil is to be burnt, connected with an aspirator and a condenser, so that the vapours are drawn off and condensed as quickly as they are formed. 70 to 90 grams at a time can be treated in this way, and when one portion has been taken down to the coking point, another can be added until 200 to 300 grams have been volatilised, when the combustion of the small residual amount of fixed products can be completed over the blow-pipe or in a stream of oxygen. The head may be improvised out of a flask with the bottom cut out.

B. B.

Chemical Researches on Sicilian Cheese. Spica and De Blasi (*Staz. Sper. Ag. Ital.*, xxiii., 133.)—The authors discuss the different kinds of Italian and Sicilian cheeses They draw attention to the fact that useful analyses are very scarce; for instance, the only analyses of "Cacio-cavallo" being those of Sartori (THE ANALYST, xviii., 17). They have, therefore, collected the results of 23 analyses made by themselves on typical cheeses: these include 12 Placentian and 2 Majorcan cheeses, and 9 of "cacio-cavallo."

Their methods are as follows:—*Preparation of the Sample.*—A square lump of about a kilogramme was cut out of the middle of the cheese; this was scraped for a thickness

of 2 c.m., and kept in a stoppered bottle; each time a portion was taken for analysis the top part was scraped off. *Water.*—5 grammes of cheese were mixed in a mortar with a glass pestle, and then dried to constant weight at 110° C. *Ash.*—5 grammes were burnt in a platinum capsule. When carbonised, the coal was washed with hot water to extract the soluble ash; the carbon was then burnt at a high temperature, and the solution added to the capsule and evaporated. *Sodium Chloride.*—The ash was lixiviated, and the chlorine, estimated by silver nitrate volumetrically, was calculated as NaCl. *Phosphates* were estimated by Spica's method (THE ANALYST, xvii., 116); the modification of adding sodium carbonate and sand, free from iron, to the cheese before incinerating, was adopted. *Fat.*—10 grammes of cheese were extracted with petroleum ether in a "Soxhlet"; the soluble acids are estimated by this method as fat. *Nitrogenous Matters.*—These were estimated as total nitrogen, proteid nitrogen, ammoniacal nitrogen, and amidic nitrogen. Total nitrogen was estimated by combustion with soda-lime; 2 grammes of starch being added to 4 grammes of cheese; proteid nitrogen was estimated by the combustion of the portion insoluble in hot water, with the addition of the lead acetate precipitate; for ammoniacal nitrogen, 5 grammes of cheese were washed on a filter with about a litre of cold water, the filtrate evaporated and distilled with 5 grammes of magnesia, and the ammonia titrated with N/10 sulphuric acid. The amidic nitrogen was estimated in the filtrate from the proteid nitrogen, after elimination of soluble proteids by precipitation with acetate of lead, the ammoniacal and nitric nitrogen, if any, being subtracted. Traces of nitrates were found in some samples of cheese. Acidity, as lactic acid, was estimated by washing 5 grammes of cheese till the washings were no longer acid, and titrating with N/10 KOH.

The means of the analyses were:—

	Placentian.	Cacio-cavallo.
Water	29·06	23·67
Ash	9·46	7·63
Fat	24·74	25·49
Nitrogenous matters ...	30·09	29·25
Proteids	23·71*	23·63
Nitrogen total ...	5·056	4·865
,, proteid ...	3·714	3·780
,, amidic ...	1·171	0·987
,, ammoniacal ...	0·091	0·097
NaCl	5·04	3·39
P_2O_5	1·197	1·278
Acidity as lactic acid ...	1·55	1·74

* Possibly a misprint for 23·21.

The most notable difference between these two types of cheese lies in the amidic nitrogen

[Note by Abstractor.—Kjeldahl's method is more suited to the analysis of cheese than that used by the authors. For much information that is not of an analytical nature, the original paper must be consulted.] H. D. R.

Standardising Acidimetric and Alkalimetric Solutions. (*School of Mines Quarterly*, xiv., 62).—Parsons concludes from his experiments that the most accurate method of standardising is with potassium tetroxalate (volumetrically). Of the gravitation methods the determination (of standard HCl) by precipitation as AgCl is given the preference. The tetroxalate as usually obtained, frequently contains more or less acid (bi) oxalate. Using an excess of oxalic acid for the first crystallization and keeping the solution hot for an hour or more before cooling to crystallize out, are especially advised. No material gain or loss by exposure to ordinary conditions, or by drying over sulphuric acid was detected. Litmus was found to be the best indicator with it. The point taken is the distinct *appearance of the blue* (adding an alkaline solution from the burette). Cochineal, methyl orange and turmeric, are unsatisfactory. Acid potassium tartrate, strongly recommended by Börntrager (*Vid. Quarterly*, xiii., 175), was found to be more difficult of preparation, as it tends to retain an excess of acid. Börntrager's directions are not sufficient for obtaining a salt of the necessary purity. W. J. S.

CORRESPONDENCE.

County Analyst's Office, Darlington,
March 10*th*, 1893.

THE STOCK NITROGEN PROCESS.

Sirs, *To the Editors of* The Analyst.

This method, if one may judge from the character of the discussion which followed the reading of my last communication upon the subject, appears to have reached a stage at which, and beyond which, no further information is to be elicited by either argument or assertion. If it needs anything more to establish its claim to a front rank among accurate methods for the determination of nitrogen in organic bodies, it needs *work*.

May I say to analysts who intend to try this new process, that the confession of a skilled and experienced operator (Mr. Allen) that he "found it desirable," in working the Kjeldahl process as now known, "to make two or three determinations and take the highest result," is in singular contrast with the fact that this same method has been selected as a standard of accuracy by those who have had most to say in criticising my process.

And may I also say that I shall be most willing to do all I can, if applied to by letter, to help others over difficulties I have myself surmounted. Yours obediently,

W. F. KEATING STOCK.